# COOK INLET SUBSISTENCE CONSUMPTION ASSESSMENT OF THE SELDOVIA, PORT GRAHAM, NANWALEK, AND TYONEK TRIBES OF COOK INLET, AK

## Phase II: Contaminant testing of Sockeye Salmon

## Quality Assurance Project Plan

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**TABLE OF CONTENTS**

## 1.0 ACKNOWLEDGEMENTS

This project is being undertaken by the Seldovia Village Tribe (SVT)'s Environmental Staff through an Environmental Protection Agency (EPA) Indian General Assistance Program (IGAP) Unmet Needs Grant. This project is part of a 2nd phase of a subsistence consumption assessment of Cook Inlet tribes (Seldovia, Port Graham, Nanwalek, and Tyonek). The 1st phase of this assessment was a survey of tribal members to determine consumption rates of subsistence foods (mainly fish and shellfish). This survey took place between November 2011 and September 2012. The 2nd phase of this assessment is tissue sampling of priority subsistence foods for contaminants. SVT was funded to conduct tissue sampling of Sockeye salmon within Cook Inlet during the summer of 2014. This will be a collaborative project amongst the four aforementioned tribes, the Alaska Department of Environmental Conservation (ADEC) through their Fish Tissue Testing (i.e. Fish Monitoring) Program, and EPA. We sincerely wish to thank EPA for funding this project, ADEC for providing free laboratory and shipping services, and our partner tribes for assisting with this project.

## 2.0 OBJECTIVES

The objective of this project is to protect and enhance the health of Cook Inlet tribal members by collecting data on contaminants present in priority fish species eaten by tribal members (specifically Sockeye salmon). Specific goals include:

1) The analyses of whole body samples of Sockeye salmon collected from Cook Inlet to determine levels of chemical contaminants present in these samples.

2) To establish a more comprehensive database of contaminant concentrations related to traditional foods harvested within Cook Inlet.

3) SVT and SVT staff will build capacity to collect contaminant data.

This QAPP is designed to ensure that all fish tissue sample analytical results are of consistent, high quality so that the best information is made available to evaluate and protect traditional resources of Cook Inlet tribal members.

## 3.0 BACKGROUND

Cook Inlet stretches 180 miles (290 km) from the Gulf of Alaska to Anchorage in south-central Alaska. This large tidal estuary covers about 100,000 km² of southern Alaska, east of the Aleutian Range and south of the Alaska Range. At least 150 rivers and streams empty into Cook Inlet. For thousands of years, native Alaskans have relied on the rich diversity and abundance of animals and plants residing in Cook Inlet as traditional foods. Development and oil and gas activities occurring in Cook Inlet have raised great concerns over contaminants in traditional foods harvested within Cook Inlet and the risk these contaminants pose to human health. Tyonek is within 10 miles from the nearest oil and gas operations while Seldovia is approximately 117 miles away and Port Graham and Nanwalek are about 128 miles away (USEPA 2000, 2003). However, this project is not trying to ascertain or link sources of contaminants present in traditional foods with potential sources nor can the data collected as a result of this project be correlated with contaminants present in Cook Inlet waters.

*PAST EVALUATIONS*

Commented [KL1]: Contaminant concentrations should be summarized by location, species, and chemical. Plots of contaminant concentrations and maps would be helpful. This approach would integrate all of the results discussed below.

Previous investigations by federal and state agencies have identified metals, pesticides, polychlorinated biphenyls (PCB), polycyclic aromatic hydrocarbons (PAH) and dioxin compounds in traditionally eaten foods from Cook Inlet. Contaminant data from tissue sampling of fish and shellfish within Cook Inlet has been previously collected through studies undertaken by multiple agencies and organizations.

These studies/projects include:

1. Fish Tissue Testing Program (i.e. Fish Monitoring Program) – ADEC:

Supported by funding from the EPA, National Oceanic and Atmospheric Administration (NOAA) and Bureau of Ocean Energy Management, Regulation, and Enforcement (BOEMRE), contaminant data for salmon (all five species), halibut, pacific cod, sablefish, black rockfish, sheefish, lingcod, pollock as well as other marine and fresh water species throughout Alaska have been collected for trace metals (methyl mercury, total mercury, selenium, copper, lead, cadmium) and organic contaminants. Most of the data specific to fish caught in Cook Inlet was collected between 2001 and 2010. More information about this program can be found at http://www.dec.state.ak.us/ADEC EH/vet/fish.htm. **The data collected in this project will be incorporated into ADEC's fish tissue monitoring program database and subsequently used for their purposes. Data collected through this program is used by ADEC to determine if 1) on-going routine sampling is needed for sentinel monitoring, 2) what areas or species that may need further evaluation, 3) new species or locations that need to be assessed, and 4) actions needed to be taken to mitigate any negative impacts of environmental pollutants on Alaska's environmental resources. This is a collaborative program and so the data is shared with university researchers, other state and federal agencies (EPA, NOAA, Department of Interior, ADF&G, DHSS) to further work in evaluating toxicologic impacts on coastal ecosystems and salmon health issues. All sampling methods and SOP's will follow ADEC's current protocols for this project.**

**Support Data for Fish Collected for Organics Analysis in the Fish Tissue Testing Program, 2001-2012**

| | Aleutians | Bering Sea | Bristol Bay | Cook Inlet | GOA | PWS | SE | FW-Arc | FW-Int | FW-SC | FW-SW |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Pacific Halibut | 17 | | | | 19 | 4 | 21 | | | | |
| Pacific Cod | 20 | | | | | | | | | | |
| Sablefish | | | | | 19 | | 20 | | | | |
| Walleye Pollock | | 4 | 12 | | | | | | | | |
| Dusky Rockfish | 15 | | | | | | | | | | |
| Capelin | | 1 | | | | | | | | | |
| Eulachon | | | | 7 | | | | | | | |
| Pacific Herring | | 1 | | 10 | | | | | | | |
| Sand Lance | | 1 | | | | | | | | | |
| Salmon Shark | | | | | 1 | 7 | | | | | |
| Sleeper Shark | | | | 1 | | | | | | | |
| Chinook Salmon | | 7 | 7 | 6 | | | 8 | | 35 | 6 | |
| Sockeye Salmon | | | 6 | | 27 | | 18 | | | 6 | 16 |
| Coho Salmon | | | | | | 11 | 107 | | | 18 | |
| Chum Salmon | | 6 | 15 | | | 2 | 26 | | 3 | | 6 |
| Pink Salmon | | | | | | 10 | 14 | | | 10 | |
| Arctic Char | | | | | | | | | | 1 | |
| Dolly Varden | | | | | | | | | | 1 | |
| Lake Trout | | | | | | | | | | | 11 |
| Humpback Whitefish | | | | | | | | | | | 2 |
| Sheefish | | | | | | | | 8 | | | |
| Northern Pike | | | | | | | | | | | 10 |

GOA: Gulf of Alaska
PWS: Prince William Sound
SE: Southeast Alaska
FW-Arc: Freshwater, Arctic
FW-Int: Freshwater, Interior (Yukon Drainages)
FW-SC: Freshwater, Southcentral Alaska, including Kodiak Island
FW-SW: Freshwater, Southwest Alaska, including Bristol Bay and Kuskokwim drainages

Non-detetects in the Organic Results Tables are treated as 1/2 the detection limit when calculating compound concentrations
Chlordanes are cis-, trans-, and oxychlordane, and cis- and trans-nonachlor
DDT is 2,4-DDD, 4,4-DDD, 2,4-DDE, 4,4-DDE, 2,4-DDT, and 4,4-DDT,
Lindane-HCH is Alpha, beta, delta, and gamma (lindane) Hexachlorocyclohexane
Toxaphene is an undefined mix of similar compounds

*Alaska Department of Environmental Conservation*
*Fish Tissue Testing Program*

In terms of Cook Inlet data, what ADEC presently has contaminant information on (as of December 2013) is:

| Heavy Metals: | | Organics: | |
|---|---|---|---|
| Species/Taxa | Years of Data | Species/Taxa | Years of Data |
| Clams | 1996-2001 | | |
| Sleeper Shark | 2013 | Sleeper Shark | 2013 |
| Pacific Cod | 2001-2009 | Pacific Cod | 2010 |
| Dolly Varden | 2008 | | |
| Eulachon | 2009 | Eulachon | 2010 |
| Grayling | 2008 | | |
| Halibut | 2002-2007 | | |
| Pacific Herring | 2008-2010 | Pacific Herring | 2008 |
| Lingcod | 2002 and 2010 | | |
| Walleye Pollock | 2002 and 2009 | | |
| Rockfish | 2007 | | |
| Chinook Salmon | 2001 and 2006 | Chinook Salmon | 2002 |
| Chum Salmon | 2002 | | |
| Pink Salmon | 2002 | | |
| Sockeye Salmon | 2002-2003 | | |
| Coho Salmon | 2002-2006 | | |
| Spiny Dogfish | 2001-2002 | | |
| Rainbow Trout | 2009 | | |

| Sample Type | Tissue | Region | N for arsenic, cadmium, lead | N for copper | N for selenium | N for mercury |
|---|---|---|---|---|---|---|
| Pacific Halibut | Fillet | Homer | 56 | 48 | 56 | 56 |
| Pacific Cod | Fillet | Homer | 45 | 34 | 45 | 45 |
| Lingcod | Fillet | Homer | 17 | 13 | 17 | 17 |
| Kelp Greenling | Whole | Homer | 5 | 5 | 5 | 5 |
| Walleye Pollock | Fillet | Homer | 14 | 3 | 14 | 14 |
| Black Rockfish | Fillet | Homer | 2 | 0 | 2 | 2 |
| Dusky Rockfish | Fillet | Homer | 3 | 2 | 3 | 3 |
| Rougheye Rockfish | Fillet | Homer | 17 | 15 | 17 | 17 |
| Yelloweye Rockfish | Fillet | Homer | 2 | 0 | 2 | 2 |
| Eulachon | Whole Composite | Kenai | 7 | 7 | 7 | 7 |
| Pacific Herring | Whole Composite | Homer | 10 | 10 | 10 | 10 |
| Starry Flounder | Whole | Homer | 1 | 1 | 1 | 1 |
| Southern Rock Sole | Whole | Homer | 1 | 1 | 1 | 1 |
| Sleeper Shark | Fillet | Homer | 1 | 1 | 1 | 1 |
| Spiny Dogfish | Fillet | Homer | 1 | 0 | 0 | 0 |
| | Fillet | Kenai | 1 | 0 | 1 | 0 |
| Chinook Salmon | Fillet | Homer | 6 | 0 | 6 | 6 |
| | Fillet | Kenai | 5 | 0 | 5 | 5 |
| Sockeye Salmon | Fillet | Homer | 6 | 0 | 6 | 6 |
| | Fillet | Kenai | 9 | 0 | 9 | 9 |
| Coho Salmon | Fillet | Homer | 6 | 0 | 6 | 6 |
| | Fillet | Kenai | 10 | 0 | 10 | 10 |
| Chum Salmon | Fillet | Homer | 6 | 0 | 6 | 6 |
| Pink Salmon | Fillet | Homer | 6 | 0 | 6 | 6 |
| Grayling | Fillet | Kenai | 8 | 8 | 8 | 8 |
| Dolly varden | Fillet | Kenai | 6 | 6 | 6 | 6 |
| Northern Pike | Fillet | Kenai | 1 | 1 | 1 | 1 |
| Rainbow Trout | Fillet | Kenai | 2 | 2 | 2 | 2 |
| Butter Clam | Whole Tissue | Homer | 16 | 0 | 0 | 0 |
| Cockle | Whole Tissue | Homer | 4 | 0 | 0 | 0 |
| littleneck clam | Whole Tissue | Homer | 29 | 0 | 0 | 0 |
| Razor Clam | Muscle Tissue | Homer | 21 | 0 | 0 | 0 |
| | Muscle Tissue | Kenai | 3 | 0 | 2 | 2 |
| | Muscle Tissue | West Cook Inlet | 17 | 0 | 0 | 0 |
| Redneck Clam | Whole Tissue | Homer | 6 | 0 | 0 | 0 |
| | Whole Tissue | Kenai | 1 | 0 | 0 | 0 |
| | Whole Tissue | West Cook Inlet | 1 | 0 | 0 | 0 |
| Softshell Clam | Whole Tissue | Homer | 2 | 0 | 0 | 0 |
| Blue Mussel | Whole Tissue | Homer | 47 | 0 | 0 | 0 |
| Pacific Oyster | Whole Tissue | Homer | 56 | 0 | 1 | 1 |
| Bay Scallop | Muscle Tissue | Homer | 9 | 0 | 0 | 0 |

| | | | | | | |
|---|---|---|---|---|---|---|
| Neptunea pribilofensis snail | Whole Tissue | West Cook Inlet | 1 | 0 | 0 | 0 |
| Octopus | Whole Tissue | Homer | 1 | 0 | 0 | 0 |

**Typical contaminant levels found in Sockeye salmon in Alaskan waters (based on ADEC fish tissue monitoring program data). ND=not detected. Data available at http://dec.alaska.gov/eh/vet/fish.htm:**

*PCBs, PBDEs, and pesticides (Concentrations in parts/billion wet weight)*

| Contaminant | Type of sample | Mean ± Std Dev | Median | Range |
|---|---|---|---|---|
| PCBs-Congener 153 | Fillet | .54 ± .46 | .30 | 0.017-2.0 |
| | Whole | .54 ± .29 | .49 | 0.15-1.3 |
| PCBs-total | Fillet | 6.8 ± 5.6 | 4.4 | 0.24-23 |
| | Whole | 7.1 ± 3.5 | 6.5 | 2.2-17 |
| PBDE-Congener 47 | Fillet | .083 ± .16 | .040 | .009-.93 |
| | Whole | .23 ± .33 | .074 | .041-1.2 |
| PBDEs-total | Fillet | .31 ± .35 | .20 | .074-2.1 |
| | Whole | .74 ± .97 | .27 | .13-3.7 |
| Pesticides-sum DDT | Fillet | 7.3 ± 5.9 | 5.0 | .17-22 |
| | Whole | 5.2 ± 2.5 | 4.9 | 1.4-9.7 |
| Pesticides-sum Chlordanes | Fillet | 10 ± 24 | 1.9 | ND-113 |
| | Whole | 1.9 ± .65 | 1.9 | .84-3.2 |
| Pesticides-total Toxaphenes | Fillet | 12 ± 10 | ND | ND-39 |
| | Whole | 16 ± 7.8 | 14 | ND-30 |
| Pesticides-Dieldrin | Fillet | .38 ± .29 | .30 | ND-1.3 |
| | Whole | .30 ± .11 | .27 | .08-.51 |
| Pesticides-Lindane and other hexachlorocyclohexane | Fillet | 1.2 ± 1.2 | .78 | .10-5.2 |
| | Whole | .85 ± .67 | .76 | .06-2.4 |
| Pesticides-hexachlorobenzene | Fillet | 1.2 ± .80 | .96 | .20-3.7 |
| | Whole | 1.4 ± .47 | 1.4 | .46-2.1 |

*Dioxins/Furans (Concentrations in parts/trillion wet weight)*

| Contaminant | Type of sample | Mean ± Std Dev | Median | Range |
|---|---|---|---|---|
| Dioxins/Furans-2,3,7,8-Tetrachloro-dilbenzo-dioxin | Fillet | ND | ND | ND-.088 |
| | Whole | .051 ± .019 | 0.059 | ND-.077 |
| Dioxins/Furans-Sum of 4 to 8 Chlorine | Fillet | 1.4 ± .94 | 1.1 | .27-4.8 |
| | Whole | 1.4 ± .76 | 1.2 | .34-3.2 |

*Heavy Metals (Concentrations in parts/million wet weight)*

| Contaminant | Type of sample | Mean ± Std Dev | Median | Range |
|---|---|---|---|---|

| | | | | |
|---|---|---|---|---|
| Total mercury | Fillet | .038 ± .014 | .038 | ND-.082 |
| | Whole | .031 ± .010 | .033 | .012-.051 |
| Total Arsenic | Fillet | .27 ± .12 | .26 | ND-.95 |
| | Whole | .28 ± .084 | .27 | .12-.43 |
| Cadmium | Fillet | ND | ND | ND-.070 |
| | Whole | .029 ± .014 | .026 | .011-.061 |
| Chromium | Fillet | ND | ND | ND-.16 |
| | Whole | .20 ± .13 | .21 | .070-.32 |
| Copper | Fillet | .67 ± .21 | .63 | .41-1.5 |
| | Whole | 5.4 ± 2.6 | 6.1 | .84-9.0 |
| Lead | Fillet | ND | ND | ND-.030 |
| | Whole | ND | ND | ND-.044 |
| Nickel | Fillet | ND | ND | ND-.29 |
| | Whole | .22 ± .064 | .23 | .13-.28 |
| Selenium | Fillet | .23 ± .061 | .23 | .090-.46 |
| | Whole | .54 ± .11 | .54 | .30-.70 |

2. **Assessment of Contaminant Body Burdens and Histopathology of Fish and Shellfish Species Frequently Used for Subsistence Food** by Chugach Native Communities (NPRB - Project-1019; July 1, 2010-February 28, 2013):

**Commented [KL2]:** What were the results?

This project was a collaborative effort amongst the Chugach Regional Resources Commission (CRRC), the Alutiiq Pride Shellfish Hatchery, the NOAA National Status and Trend (NS&T) Program, and the Northwest Fishery Science Center (NWFSC). This study assessed the contaminant status and histopathology condition of two species of salmon (chum and Sockeye salmon) and shellfish (cockles and littleneck clams) commonly harvested by natives in the Chugach region. The fish and shellfish were collected from traditional subsistence harvest areas in the vicinity of Nanwalek, Port Graham and Seldovia. Tissue was analyzed for trace metals and residues of organic contaminants routinely monitored by NS&T program, and histologically characterized for the presence, prevalence and severity of tissue pathology, disease, and parasite infection.

3. **Evaluation of seafood and plant data collected from Cook Inlet near the native villages of Port Graham, Nanwalek, Seldovia, and Tyonek, Alaska**- Agency for Toxic Substances and Disease Registry (ATSDR)-2009:

**Commented [KL3]:** What were the results?

EPA collected whole fish, mussels/clams, other invertebrates (i.e. snail, chiton, and octopus) and plants from Cook Inlet in 1997. Between June and August 2002, ADEC collected 65 fish (as part of their Fish Tissue Testing Program) that included Pacific cod, chinook salmon, pink salmon, chum salmon, red salmon, silver salmon, pollock, and halibut from lower Cook Inlet. Skinless fillets and halibut roast from 47 fish were analyzed for heavy metals. Fillets from six Chinook salmon were also analyzed for pesticides, dioxins, and polychlorinated biphenyls (PCBs).

4. **Cook Inlet Regional Citizens Advisory Council (CIRCAC) Environmental Monitoring Program** – 1993, 1996, and 2000:

**Commented [KL4]:** What were the results?

Beginning in 1993, CIRCAC began a series of preliminary studies to assess impacts of oil and gas operations on Cook Inlet. In 1993 and 1996, total polycyclic aromatic hydrocarbons (PAHs) were measured in mussels and deposit-feeding clams from seven locations in Cook Inlet and one

location in Shelikof Strait. In 2000, PAH concentrations were measured in 3 razor clams, 2 mussels, and 3 deposit-feeding clams from the east side of upper Cook Inlet; 4 soft shell clams, 1 razor clam, and 2 deposit-feeding clams from the middle of upper Cook Inlet; and 5 deposit-feeding clams, 1 mussel, 2 razor clams, and 1 softshell clam from the west side of upper Cook Inlet.

**See Appendix I for map of sampling locations of these past studies.**

Contaminants within the water column and sediments of Cook Inlet have also been examined. These contaminants can subsequently influence contaminants found in traditional foods harvested from Cook Inlet waterways although this would not necessarily be applicable to non-resident fish species, like the red salmon being tested in this project. Potential reference sources for these contaminant data are:

1. Hartwell, S.I., Apeti, D., Claflin, L.W., Johnson, W.E. and Kimbrough, K. 2009. Sediment Quality Triad Assessment in Kachemak Bay: Characterization of Soft Bottom Benthic Habitats and Contaminant Bioeffects Assessment. NOAA Technical Memorandum NOS NCCOS 104. 170pp. (NPRB - Project 726; 7/1/2007-10/30/2009)

2. Pollution and Biological Health Assessment of Fjords on Kenai Peninsula, Alaska

NOAA/NCCOS Project Status: This project began in August 2009 and is still ongoing

This study builds on the National Status and Trends (NS&T) bioeffects assessment of the northern side of Kachemak Bay, completed in 2007, and an assessment in the deep central portion of Kachemak Bay, conducted in 2008 in collaboration with the Cook Inlet Regional Citizens Advisory Council (CIRCAC). It is a joint project with the Alaska Department of Environmental Conservation.

A baseline environmental characterization was done of the fjords and embayments along the south shore of Kachemak Bay and the outer Kenai Peninsula using the sediment quality triad approach. The triad includes: sediment chemistry, sediment toxicity, and benthic invertebrate community structure. Concentrations of over 120 organic and metallic contaminants are being analyzed. Sediment toxicity is being assessed using amphipod bioassays with sediment from the abandoned mine sites. Fish and mussels from selected locations are undergoing contaminant body burden analyses.

Joint field operations were completed in 2009 with the assistance of the NOAA Kasitsna Bay Laboratory and the Kachemak Bay Estuarine Research Reserve.

Thus far, they have found that organic contaminants were elevated in the vicinity of Seldovia Harbor.

**Commented [KL5]:** Basis for conclusion? Was the elevation in contaminant concentrations significant?

3. Frenzel, S.A. 2000. Selected Organic Compounds and Trace Elements in Streambed Sediments and Fish Tissues, Cook Inlet Basin, Alaska. U.S. Geological Survey, Water-Resources Investigations Report 00-4004.

4. Henrichs, S.M., Schell, D.M., Borland, T., Howe, T. 2003. Hydrocarbon sources in Kachemak Bay Sediments: Improved Discrimination by Specific Compound $\delta$ 13C Measurements. Institute

of Marine Science, School of Fisheries and Ocean Sciences, University of Alaska Fairbanks. Final Report submitted to the NOAA/UNH Cooperative Institute for Coastal and Estuarine Environmental Technology (CICEET).

5. Saupe, S.M., J. Gendron, and D. Dasher. 2005. The Condition of Southcentral Alaska Coastal Bays and Estuaries. A Statistical Summary for the National Coastal Assessment Program Alaska Department of Environmental Conservation, MARCH 15, 2006.

6. Segar, D.A. 1995. Current water quality in Cook Inlet, Alaska, Study. Environment and Natural Resources Institute, University of Alaska Anchorage. Report for U.S. Department of the Interior, Mineral Management Services, OCS Study MMS 95-0009.

7. Kinnetic Laboratories, Inc. 2004. Knik Arm Crossing Preliminary Offshore Water Quality Assessment Technical Memorandum. Report for Knik Arm Bridge and Toll Authority, Alaska Department of Transportation and Public Facilities, Federal Highway Administration, and HDR Alaska, Inc.

*8. SVT – 2005*. Indian General Assistance Program (IGAP) funded work. Data/Results summarized in SVT's summer 2005 Baseline Sampling Report.

In 2005, SVT environmental staff collected water and sediment samples from sites within Seldovia Bay as part of IGAP funded work. Seldovia Bay Sites consisted of sites along transect lines, four benthic sites, four sewage discharge localized sites and three land fill seepage localized sites. The four transects (T1, T2, T3 and T4) were logically placed in Seldovia Bay to represent the outer bay interface with Kachemak Bay (T1), the outer third of the bay (T2), the mid bay (T3) and the inner bay and interface with the flats and bay's primary fresh water sources. Each of these transect lines had three stations located along their axis. Benthic sites (B1, B2, B3 and B4) were also oriented from the outer bay towards the inner bay. These benthic sites where all either defined sinks or localized deeper spots that were likely to contain trapped sediments. The other sites were issue oriented sampling specific sites including the sewage discharge line (SD1, SD2, SD3 and SD4) moving away from and into the bay from the discharge out fall pipe (SD1) and then Dan's Cove which is a small cove with a small stream running out from below an old dump site (DC1, DC2 and DC3).

**Commented [KL6]:** Definition?

The following table shows the actual chemical sampling events in chronological order by date which were sent in to the laboratory, Analytica, for analysis of polycyclic aromatic hydrocarbons (PAH's), volatile organic compounds, and metals (Arsenic, Barium, Cadmium, Chromium, Lead, Selenium, Mercury). Both water and sediment samples were analyzed.

**Commented [KL7]:** Would be helpful to maps showing concentrations of specific contaminants.

| Lab Sample Number | Client Description | Matrix | Date Sampled | Time Sampled | Date Received |
|---|---|---|---|---|---|
| A0508057-01 | TIS SW | Salt Water | 3-Aug | 9:07 AM | 4-Aug |
| A0508057-02 | TID SW | Salt Water | 3-Aug | 9:30 AM | 4-Aug |
| | | | | | |
| A0508079-01 | T4S | Salt Water | 4-Aug | 10:52 AM | 5-Aug |
| A0508079-02 | B2D | Salt Water | 4-Aug | 9:22 AM | 5-Aug |
| A0508079-03 | B1D | Salt Water | 4-Aug | 9:00 AM | 5-Aug |
| A0508079-04 | B4S | Salt Water | 4-Aug | 10:03 AM | 5-Aug |
| A0508079-05 | B3S | Salt Water | 4-Aug | 9:41 AM | 5-Aug |
| A0508079-06 | B3D | Salt Water | 4-Aug | 9:52 AM | 5-Aug |
| A0508079-07 | B2S | Salt Water | 4-Aug | 9:12 AM | 5-Aug |
| A0508079-08 | B4D | Salt Water | 4-Aug | 10:16 AM | 5-Aug |
| A0508079-09 | T4D | Salt Water | 4-Aug | 10:45 AM | 5-Aug |
| A0508079-10 | B1S | Salt Water | 4-Aug | 8:45 AM | 5-Aug |
| A0508079-11 | DC SW | Salt Water | 4-Aug | 11:09 AM | 5-Aug |
| A0508079-12 | DC FW | Fresh Water | 4-Aug | 11:00 AM | 5-Aug |
| | | | | | |
| A0508097-01A | SD1 Outfall Pipe | Salt Water | 8-Aug | 9:20 AM | 8-Aug |
| A0508097-02A | SD 2 MZ | Salt Water | 8-Aug | 9:07 AM | 8-Aug |
| A0508097-03A | SD 3 | Salt Water | 8-Aug | 10:10 AM | 8-Aug |
| A0508097-04A | SD 4 | Salt Water | 8-Aug | 10:38 AM | 8-Aug |
| A0508097-05A | SD 4 | Sediment | 8-Aug | 10:43 AM | 8-Aug |
| A0508097-06A | SD 1 Outfall Pipe | Sediment | 8-Aug | 9:55 AM | 8-Aug |
| A0508097-07A | SD 3 | Sediment | 8-Aug | 10:22 AM | 8-Aug |
| A0508097-08A | SD 2 | Sediment | 8-Aug | 9:22 AM | 8-Aug |
| | | | | | |
| A0508149-01 | B1 | Sediment | 10-Aug | 9:15 AM | 10-Aug |
| A0508149-02 | SD # 2 | Sediment | 10-Aug | 10:56 AM | 10-Aug |
| A0508149-03 | B3 | Sediment | 10-Aug | 11:26 AM | 10-Aug |
| A0508149-04 | B4 | Sediment | 10-Aug | 11:49 AM | 10-Aug |
| A0508149-05 | DC | Sediment | 10-Aug | 12:10 PM | 10-Aug |
| A0508149-06 | DC2 | Sediment | 10-Aug | 12:35 PM | 10-Aug |
| A0508149-07 | DC3 | Sediment | 10-Aug | 12:42 PM | 10-Aug |
| | | | | | |
| A0508234-01 | B1 Shallow | Salt Water | 18-Aug | 8:55 AM | 19-Aug |
| A0508234-02 | B1 | Sediment | 18-Aug | 9:06 AM | 19-Aug |
| A0508234-03 | B2 | Sediment | 18-Aug | 9:40 AM | 19-Aug |
| A0508234-04 | B3 | Sediment | 18-Aug | 10:40 AM | 19-Aug |
| A0508234-05 | B4 | Sediment | 18-Aug | 11:37 AM | 19-Aug |
| A0508234-06 | DC | Sediment | 18-Aug | 10:57 AM | 19-Aug |
| A0508234-07 | DC2 | Sediment | 18-Aug | 11:55 AM | 19-Aug |
| A0508234-08 | SD SW | Salt Water | 18-Aug | 11:08 AM | 19-Aug |
| A0508234-09 | Trip Blank | Water | 18-Aug | | 19-Aug |
| A0508234-10 | SD | Sediment | 18-Aug | 11:18 AM | 19-Aug |
| A0508234-11 | DC3 | Sediment | 18-Aug | 12:16 PM | 19-Aug |
| | | | | | |
| Sediment Metals | | | | | |
| A508156 | B1 | Sediment | 10-Aug | 9:15 AM | 16-Aug |
| A508156 | B3 | Sediment | 10-Aug | 11:26 AM | 16-Aug |
| A508156 | B4 | Sediment | 10-Aug | 11:49 AM | 16-Aug |
| A508156 | DC | Sediment | 10-Aug | 12:10 PM | 16-Aug |
| | | | | | |
| A508240 | SD | Sediment | 18-Aug | 11:18 AM | 24-Aug |
| A508240 | B2 | Sediment | 18-Aug | 9:40 AM | 24-Aug |

*PRESENT CONCERNS*

Based on existing data, levels of chemicals found in many native foods from Cook Inlet appear to be often at levels that are found in fish from other parts of Alaska or from grocery stores (ATSDR 2009, Apeti et al. 2013, ADEC Fish Monitoring Program data). Additionally, in general, Sockeye salmon in Alaska waters appear to have contaminant levels around, or below, those found in salmon within the Columbia River Basin (see below table). However, before comparing data between different studies, caution should be used to determine that equivalent analytical methods were used. **For this project, existing methods established and utilized by the ADEC Fish Tissue Monitoring Program will be used and the data incorporated into their existing database.**

Range of chemical concentrations found in salmon in Columbia River Basin fish tissue samples (whole-body; based on wet weight). Data found in US EPA. 2002. Columbia River Basin Fish Contaminant Survey 1996-1998. EPA Report 910-R-02-006:

**Commented [KL8]:** If a chemical can be detected at "natural" background levels, then ADEC reporting limits are fine. Were there any contaminants that could not be detected? If so, what would a risk based analytical concentration goal be? Are there any methods that could attain that concentration?

**Commented [KL9]:** Why is this here?

| | Fall Chinook | | Spring Chinook | | Coho | |
|---|---|---|---|---|---|---|
| Contaminant | ug/kg | ppm | ug/kg | ppm | ug/kg | ppm |
| Total Arsenic | 610-1000 | .61-1 | 570-1100 | .57-1.1 | 450-560 | .45-.56 |
| Cadmium | 5-10 | .005-.01 | 6-170 | .006-.17 | 19-27 | .019-.027 |
| Copper | 1000-14000 | 1-14 | 1100-2300 | 1.1-2.3 | 720-2400 | .72-2.4 |
| Lead | 11-1200 | .011-1.2 | <10-92 | <.01-.092 | 11-20 | .011-.02 |
| Mercury | <50-200 | <.05-.2 | <71-130 | <.071-.13 | 11-20 | .011-.02 |
| Selenium | <380-570 | <.38-.57 | 360-680 | .36-.68 | 330-420 | .33-.42 |

| | Fall Chinook | | Spring Chinook | | Coho | |
|---|---|---|---|---|---|---|
| Contaminant | ug/kg | ppb | ug/kg | ppb | ug/kg | ppb |
| p,p'-DDE | 5-53 | 5-53 | 11-22 | 11-22 | 31-37 | 31-37 |
| p,p'-DDT | <2-7 | <2-7 | 3-8 | 3-8 | <2-4 | <2-4 |
| Arochlor 1254 | 10-47 | 10-47 | 13-26 | 13-26 | 18-19 | 18-19 |
| Arochlor 1260 | <19 | <19 | <18 | <18 | <18 | <18 |

| | Fall Chinook | | Spring Chinook | | Coho | |
|---|---|---|---|---|---|---|
| Contaminant | ug/kg | ppt | ug/kg | ppt | ug/kg | ppt |
| 2,3,7,8-TCDD | <0.0000 - 0.00006 | <0 to .06 | <0.00001 - 0.0001 | <.01-.1 | <0.00001 | <.01 |
| 2,3,7,8-TCDF | 0.00043-0.0014 | .43 – 1.4 | 0.00057 - 0.0011 | .57-1.1 | 0.00036-0.00049 | .36-.49 |

However, large gaps presently exist amongst years that contaminant data were collected for individual fish species through ADEC's Fish Tissue Monitoring Program and the other studies mentioned above, sample sizes and data are limited for particular species (especially Sockeye salmon), and previous investigations did not always target whole body fish samples or specific fish organs/portions eaten in traditional subsistence diets. Additionally, an assessment of subsistence consumption rates (fish and shellfish) of Cook Inlet tribal members from Seldovia, Port Graham, Nanwalek, and Tyonek (conducted between 2011 and 2012) undertaken by SVT, revealed that tribal members consume a much larger amount of fish per day than what is used and/or recommended by state and federal agencies to establish water quality standards in Alaska based on human health criteria (6.5 g/d and 17.5 g/d respectively). Based on the 95$^{th}$ percentile value of fish consumption rates obtained from the survey SVT undertook, SVT has recommended that a fish consumption rate of 247 g/d be used to establish water quality standards in Alaska based on human health criteria instead of the current 6.5 g/d. This implies that contaminants present in Alaskan/North Pacific waters, and subsequently in the foods eaten in traditional subsistence diets, may be having a much more significant impact on the health of tribal members than previously thought. "Tighter" water quality standards may be required to protect the health of Cook Inlet tribal members.

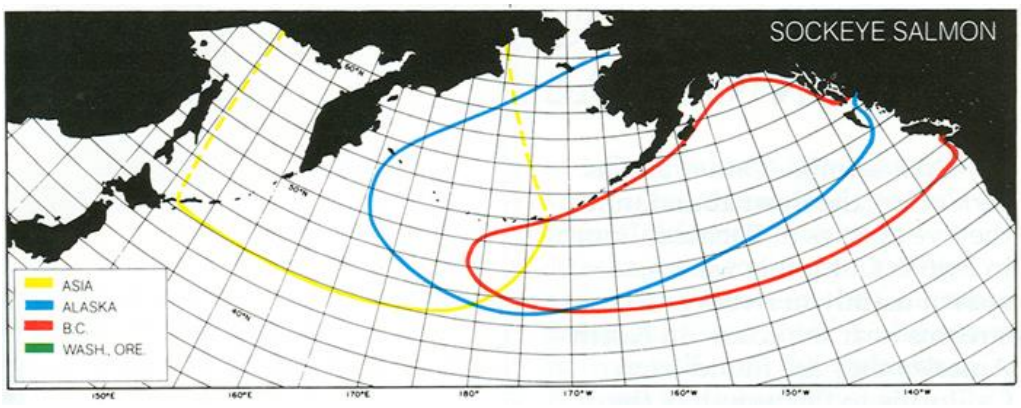
**Commented [KL10]:** Important, but not clear as to why consumption rates are being discussed in a summary of chemical contamination data available for Cook Inlet.

SVT wishes to obtain more current contaminant data for priority subsistence foods of Cook Inlet

tribal members given the aforementioned concerns. **The scale of this particular project was limited to only Sockeye Salmon and a relatively small number of samples due to budget and laboratory staff constraints ADEC is faced with. Sockeye salmon was chosen based on the high priority as a food source for Cook Inlet tribal members and the lack of data the ADEC fish tissue monitoring program staff have for contaminant data (especially organic contaminants) in regards to this species. ADEC is providing the shipping and analyses of all samples for free to SVT, enabling this project to be feasible with the funding of the IGAP Unmet Needs grant award.** Based on the assessment completed by SVT in 2012 (see reference SVT 2013), Sockeye salmon was determined to be one of the top fish species eaten by Cook Inlet tribal members. Given the importance of Sockeye salmon in traditional native diets and the limited amount of contaminant data available for this species within Cook Inlet, SVT will undertake tissue sampling of 36 whole body Sockeye salmon (harvested within Cook Inlet) for analysis of several contaminants in the summer of 2014.

As evident from the data provided by ADEC fish tissue monitoring program staff, there is a significant gap in contaminant data (especially for organic contaminants) for Sockeye salmon within Cook Inlet. To our knowledge and based on the information provided by ADEC, the last contaminant data collected for Sockeye salmon within Cook Inlet was between 2002-2003 as part of the ATSDR study Seldovia, Port Graham, Nanwalek, and Tyonek participated in and this was very limited. **ADEC's fish tissue monitoring program is supported by funding from EPA, NOAA and BOEMRE (formerly MMS) and the data collected through this program allows ADEC to identify within Alaska waters where: 1) on-going routine sampling is needed for sentinel monitoring, 2) areas or species that may need further evaluation, 3) new species or locations that need to be assessed, and 4) actions needed to be taken to mitigate any negative impacts of environmental pollutants on Alaska's environmental resources. This is a collaborative program and so the data is shared with university researchers, other state and federal agencies (EPA, NOAA, Department of Interior, ADF&G, DHSS) to further work in evaluating toxicologic impacts on coastal ecosystems and salmon health issues. The data collected as a result of SVT's involvement in this project will not only help tribes, state, and federal agencies acquire more data on contaminant levels of Sockeye salmon (and fish in general) within Cook Inlet (particularly in regards to organic contaminants), but these important data can be used to examine contaminant levels at regional and global levels amongst salmon populations and to better understand the implications thereof.**

Although outside the scope and feasibility of this project, future studies may want to examine contaminants in resident fish species or in salmon juveniles within Cook Inlet since their contaminant body burdens would be influenced by exposure to local contaminant sources and would be site specific. For instance, adult Sockeye salmon migrate from a marine environment into freshwater streams and rivers or lakes of their birth in order to mate. With the exception of certain river-type and sea-type populations, the vast majority of Sockeye salmon spawn in or near lakes, where the juveniles rear for 1 to 3 years prior to migrating to sea. For this reason, the major distribution and abundance of large Sockeye salmon stocks are closely related to the location of rivers that have accessible lakes in their watersheds for juvenile rearing. Most Sockeye salmon stay at sea for 2 years, returning to spawn at about age 4, but some may be 5-6 years old when they spawn.




This project will be undertaken as a collaborative effort between Seldovia Village Tribe, the Port Graham Tribal Council, the Nanwalek IRA Council, the Native Village of Tyonek, EPA, and the ADEC. These data will add to pre-existing databases of contaminant concentrations found within fish and shellfish collected within Cook Inlet. Such databases are important resources when state and federal agencies are considering issuing, updating, or removing human health fish consumption advisories, when undertaking environmental impact assessments, and when establishing or updating water quality standards.

**4.0 DESCRIPTION OF WORK TO BE PERFORMED**

- Collaborate with the ADEC, the Nanwalek IRA Council, the Port Graham Tribal Council, and the Native Village of Tyonek
  - Correspond with partner tribes and ADEC through teleconference calls and e-mail to keep updated with progress. Schedule planning meetings as needed
  - Develop QAPP and send to partner villages, ADEC, and EPA for comments, edits, and approval
  - Develop and post job description to hire samplers from each participating village
  - Hire two samplers who are fishing subsistence nets or have personal use fishing permits, commercial fishing licenses, and/or sport fishing licenses from each village
  - Samplers trained in proper collection and quality control techniques
  - SVT Environmental Coordinator and Assistant will travel to each village, during sampling/collection events, to oversee project activities and ensure proper preparation and shipping procedures are followed for transport of fish
  - Share findings/results with EPA, ADEC, and partner villages
- Collect 36 whole-body (WB) Sockeye salmon within Cook Inlet in the summer of 2014
  - Purchase plastic leak proof/fish bags
  - Collect a total of 9 Sockeye salmon specimens from around each participating village (Seldovia, Port Graham, Nanwalek, and Tyonek) during Sockeye runs at twp different times in the summer of 2014 (towards the middle and end of the runs). For each village, 6 fish specimens will be collected during the 1st sampling event and another 3 during the 2nd sampling event. For each village, per each sampling event, three whole body fish collected will be homogenized into one composite sample. So, for the 1st sampling event, two composite samples will be analyzed for each village along with 1 composite sample for the 2nd sampling event (or 12 composite samples

will be analyzed in total from 36 fish collected in total). The sampling design of this project is solely reflective of when the targeted fish species is locally available and when they are being harvested by the communities, the number of fish specimens ADEC can handle processing at one time, and where fish are harvested from that are consumed by tribal members in the four villages. Given the limited scale and scope of this project, it is not the intent of SVT staff to make any conclusions regarding comparisons between villages or between run periods with the data.

- Upon collection:
    - Fish, will be immediately, and individually, placed into labeled plastic leak proof/fish bags (labeled on outside of bag) using fresh nitrile gloves
    - Once the fish are placed into fish bags, fish bags will immediately be kept on ice in a cooler until arriving back on shore and then frozen at -20°C or -4°F. Thermometers will be provided and used to ensure proper temperature.
    - Fish sampling forms used by ADEC for Fish Tissue Testing Program will be filled out by sampler(s) and included with specimens
    - Labels made out of write in the rain paper will be placed inside small ziplock bags which will be placed inside fish sampling bags to reduce any potential contamination or degradation in regards to readability of labels.
- Provide data that represents areas where target fish species is harvested and consumed from
- Provide data that may be used by ADEC Fish Tissue Monitoring Program Staff Prepare and ship fish (within 24 hours) to ADEC's Environmental Health (ADEC EH) Laboratory for analysis following proper procedures

- Once fish have arrived at the ADEC EH Laboratory:
    - All fish will be stored at -20°C or -4°F or below until they are ready to be thawed for processing
    - Fork length, weight, and sex will be recorded
    - All fish will be thawed at 4°C or 39.2ºF, cut into strips, and homogenized in a tissue grinder Homogenate is then divided into 4 oz sample jars for analysis
    - Otoliths are removed from the fish for age determination
    - For each village, three whole body fish collected per sampling event, will be homogenized into one composite sample and analyzed for contaminants. Therefore, for each village, 2 composite samples will be analyzed from 6 fish collected from the 1st sampling event and 1 composite sample analyzed from 3 fish collected from the 2nd sampling event.
    - All sample jars containing samples are stored at -20°C or -4°F or below until they are analyzed and/or shipped to sub-contracted laboratory (AXYS laboratory (for organic contaminants being tested))
    - All samples analyzed for contaminants (see attached Appendices B, C, E-G)
- Samples will be analyzed for the following contaminants:
    - Polychlorinated biphenyls (PCBs)
    - Organochlorine pesticides
    - Flame-retardant Polybrominated Diphenyl Ethers (PBDEs)
    - Heavy metals (mercury, arsenic, cadmium, copper, lead, and selenium)
- Conduct data review, evaluation, and analysis
    - Mean values will be reported for contaminants within ± 2 standard deviations which provides variance around the mean equivalent to 95% Confidence Intervals. The sample variance is calculated by the following formula:

**Commented [KL11]:** It is good to acknowledge what can't be done with the data. However, given the effort being put forth, it is unfortunate that a few more samples can't be taken to get at variability across seasons and geographic locations.

**Commented [KL12]:** Note here that (hopefully…) lipids are being analyzed for as well. This appears to be the case from material presented later in the document. Lipid concentrations will affect levels of bioaccumulative contaminants and could be different for early vs. late runs.

$$s^2 = \frac{1}{N-1}\sum_{i=1}^{N}(x_i - \overline{x})^2$$

Where:
$s^2$ = sample variance
$x_1, ..., x_N$ = the sample data set
$\bar{x}$ = mean value of the sample data set
N = size of the sample data set

Sample standard deviation = square root of the sample variance or $\sqrt{s^2}$

For each contaminant tested for, a single contaminant value (i.e. a mean value) will come from every 3 fish homogenized. Every village, therefore, will have 3 values for each contaminant from the total of 9 individual fish collected (or 3 composite samples subsequently analyzed) from around each village. Per contaminant, a mean will be taken of these 3 values and reported for each village along with ± 2 standard deviations. Per contaminant, mean values will also be calculated and reported along with ± 2 standard deviations for all of the villages combined (in other words, a mean of 12 values).

- Prepare reports
- Prepare 1 page success story

**5.0 SAMPLING DESIGN AND METHODS**

The fish collection methods described in this section are intended to provide standardized, reliable, and repeatable results. Additionally, these methods are consistent with methods utilized by ADEC in their Fish Tissue Testing Program and were derived from ADEC.

5.1 Field Crew

SVT's Environmental Assistant will serve as Project Manager and be responsible for carrying out project activities. The Environmental Assistant will report to the Environmental Coordinator. Assurance oversight of grant requirements and project management are responsibilities of SVT's Environmental Coordinator and SVT's President/CEO ensures project compliance to the EPA and other regulatory agencies. In the four villages, two local residents will be hired to collect fish specimens. SVT's Environmental Assistant and Coordinator will travel to each of the partner villages, at least for the 1st collection event, to ensure samplers are following proper procedures and quality assurance/quality control is maintained.

5.2 Field Operations Schedule

Field work described in this QAPP is expected to take place during the summer of 2014. A total of 9 whole body (WB) Sockeye salmon will be collected from around each participating village (Seldovia, Port Graham, Nanwalek, and Tyonek) during Sockeye runs at two different times in the summer of 2014 (at the middle and towards the end of the runs). Six fish will be collected from around each village during the 1st sampling event and 3 fish during the 2nd sampling event. In

total, 36 WB Sockeye salmon will be sent to ADEC for analysis of contaminants. The sampling design of this project is solely reflective of when the targeted fish species is locally available and being caught by the communities, the number of fish specimens ADEC can handle processing at one time, and where fish are harvested from that are consumed by tribal members in the four villages. Given the limited scale and scope of this project, it is not the intent of SVT staff to make any conclusions regarding comparisons between villages or between run periods with the data.

**Commented [KL13]:** Or just note that it will be difficult to determine if there are any differences given such a low sample number.

Based upon typical timing runs of Sockeye salmon around each village, sampling/collection events for each village are anticipated to take place:

1st sampling event (mid summer): mid-July

2nd sampling event (late summer): mid-August

Adjustments to sampling dates may be necessary to account for variable conditions such as inclement weather, difficulties in accessing sampling locations, time needed to collect the fish, and Sockeye run times.

5.3 Sampling Location Selection Procedure

Fishing sites will be within 100 miles from each village. Based on survey information collected between 2011-2012, the vast majority of community members living in Seldovia, Tyonek, Port Graham, and Nanwalek fish within 25 miles of their respective villages. Sampling locations will be solely chosen based on local knowledge of where the target fish species can be found and are typically harvested from and not based on proximity or distance away from potential point sources of contaminants in Cook Inlet.

5.4 Sampling Gear

Gill or set nets with mesh sizes appropriate, and legal, for catching adult Sockeye salmon will be used to avoid excessive sampling effort and minimize by-catch of smaller fish. The gill nets and supporting lines will be constructed of non-tarred monofilament or twine to avoid contamination with petroleum-based compounds.

Gear and equipment required for every sampling event is provided in the table below:

| **Equipment and Supply List for Onboard Fish Collection Activities** | |
|---|---|
| Equipment | Minimum Quantity |
| Sampling vessel (including boat, motor, oars, fuel, adequate lighting and required safety equipment) | 1 |
| Gill nets (anchors, depth adjustment lines, and floats) | 1 |
| U.S. Coast Guard-approved personal floatation devices | 4 |
| Maps of sampling areas and sites | 1 |
| Nitrile gloves | 6 pairs |
| GPS unit | 1 |

| | |
|---|---|
| Ice chest | 1 |
| Buckets | 1 |
| Bags of ice | 5 |
| Fish bags | 3 |
| Labels on write in the rain paper | 3 |
| Copy of QAPP | 1 |
| Fish sampling forms | 3 |
| Sharpies, pens, and pencils | 2 of each |
| Fishing licenses/permits | 2 |
| First aid kit | 1 |
| Marine-band radio | 1 |
| Cell phone | 1 |
| Camera | 1 |

5.5 Fish Collection Procedure

From each village, two "samplers" (locally hired) will be responsible for collecting the Sockeye salmon from their surrounding fishing area(s) (see Section 5.3). At least one of the samplers hired per village must own, or have access to, a boat and be familiar with how to operate it. Both samplers must have fishing skills, knowledge and have a subsistence net they are fishing or a personal use fishing permit, commercial fishing license, and/or sport fishing license.

SVT's Environmental Coordinator or Assistant will travel to participating villages during sampling events and oversee activities, including accompanying samplers on the boat.

In general, gill nets will be set with local knowledge of when the fish are running and best times to set and pick during the time the Environmental Coordinator or Environmental Assistant are present to help collect the fish. Placement of gill nets at each site will be determined based on local knowledge of the targeted species and the site characteristics. Global positioning system (GPS) coordinates will be recorded for the specific locations where each gill net is deployed. Fish will be immediately removed from the nets as the nets are pulled into the boat, Sockeye salmon retained, and non target species kept, released, or disposed of in accordance with samplers' fishing licenses/permits and/or any other local fishing regulations they are subject to. For each village, per sampling event, only the specified number of Sockeye salmon (6 for the 1st sampling event and 3 for the 2nd) will be shipped to ADEC's laboratory and these specimens will be kept and stored separate from other fish samplers might keep. Samplers will wear fresh nitrile gloves when bagging the fish specimens. Special care will be taken to ensure that petroleum products such as grease or fuel do not come in direct contact with the fish specimens or with surfaces that contact the fish specimens.

Salmon very from stream to stream in length and weight. In Alaska, Sockeye salmon vary in length from 18 to 31 inches and weigh between 4 to 15 pounds. Kenai River Sockeye are much bigger than Nanwalek Sockeye and Nanwalek Sockeye are bigger than the Tutka Bay Hatchery Sockeye. Most people who catch Sockeye salmon can tell the difference in where the fish are from that they are catching. Hatchery fish will also not have a dorsal fin. Hatchery fish will not be sent to ADEC for analysis. Sockeye salmon will be shipped to the ADEC EH Laboratory as whole body specimens. All Sockeye salmon used as specimens will be 18" or longer, thus reducing the likelihood of getting a jack salmon. Because whole body fish will be homogenized into composite samples and results are intended to be reflective of what tribal members are consuming, it is

expected that sizes of fish will vary and that individual specimens will not contribute equally in terms of volume/weight to the composite samples.

Following is an overview of the fish collection procedures:

1. Transport sample equipment and samplers by boat to sampling locations
2. Deploy and retrieve sampling gear
3. Upon collection, fish will be immediately transferred from sampling gear to fish bags (using nitrile gloves). Filled fish bags will be kept in a cooler containing ice
4. Prepare and complete field sampling records and documentation that will be enclosed in zip lock bags and put inside the cooler
5. Put labels made out of write in the rain paper inside small zip lock bags inside fish bags and label outside of bags with permanent marker. This technique has been working for DEC fish tissue program and there has been no problems related to readability of labeling.
6. Return to shore and store specimens (in their bags) in freezer until frozen. Specimens will be stored frozen @ -20°C or -4°F. Thermometers will be provided and used to ensure proper temperature.
7. Ship specimens to laboratory for analysis within 24 hours

5.6 Labels and Field Documentation

Labels will be made from write in the rain paper and contain:
1) Sample number
2) Sample Date
3) Species
4) Location (lat and long)
5) Site Name
6) Sampler

Labels will be filled out in pencil and placed inside small zip lock bags inside fish bags with specimens.
Additionally, for each collection event, ADEC's fish sampling form will be filled out, enclosed in a zip lock bag, and then placed in the cooler with fish for shipping. Information included in the fish sampling form is as follows:
1) Sample Number
2) Sample Date
3) Species
4) Location (lat and long)
5) Site Name
6) Sampler Affiliation
7) Lead Sampler Signature

A copy of the Fish Sampling Form is provided below.

**FISH SAMPLING FORM**
**ADEC FISH TISSUE TESTING PROGRAM 2013**

| Sample # | Sample Date | Species | Location Lat | Location Long | Site Name | General Area |
|---|---|---|---|---|---|---|
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| notes: | | | | | | |
| | | | | | | |
| | | Sampler Affiliation | | | | Lead Sampler Signature |

**SAMPLING PROTOCOL**

| |
|---|
| Wear fresh gloves to bag each sample in zip-lock bags |
| Label samples consecutively by marking on sample bag |
| Place bagged samples on ice until freezing or shipment |
| Put Sample Collection Form in a zip-lock bag with the samples |
| Check with DEC Environmental Health Lab (howard.teas@alaska.gov, 907-375-8211) for shipping information |
| Take care to ensure that samples aren't exposed to exhaust or gasoline during sample collection |
| Collect as much location information as possible; Lat/long, ADF&G Sub-area, nearest point of land, or general description, as available |
| General Areas: BS (Bering Sea), GOA (Gulf of Alaska), SE (Southeast), Aleutians, Bristol Bay, PWS, Cook Inlet |
| If possible, give latitude and longitude in decimal degrees |

5.7 Storage and Transfer of Fish for Laboratory Analysis

After a collection event, fish specimens will be stored in a cooler (containing ice) and immediately upon returning to shore, the fish bags (containing the specimens) and bags of ice will be immediately placed into a freezer. Specimens will be frozen -20°C or -4°F or below (thermometers will be provided and used to ensure proper temperature) and shipped on ice in a cooler to the ADEC EH Laboratory within 24 hours of the collection date/time. All fish will be checked and identified upon arrival and frozen (-20 C or -4°F). Coolers will be labeled, sealed, and shipped as directed by ADEC staff involved in the Fish Tissue Testing Program.

5.8 Chain-of-Custody Forms

Fish Sampling Forms will serve as Chain-of-Custody Forms (as approved by ADEC) in regards to specimens being transferred from villages to ADEC EH Laboratory. Separate Chain-of-Custody forms will be used for transfer of samples from ADEC EH Laboratory to AXYS laboratory (see Appendix D)

5.9 Laboratory Analysis (see Appendices B, C, E-G)

*Processing*

All specimens will be shipped from villages directly to the ADEC EH Laboratory, whose staff will then ship samples to AXYS accordingly. Samples will be analyzed for heavy metals at the State of Alaska (ADEC)'s Environmental Health (ADEC EH) Laboratory in Anchorage, Alaska, and samples will be sent to AXYS Analytical Services in Sydney, B.C., for testing of organic contaminants (209 PCB congeners, Organochlorine pesticides (29 pesticides), and 46 PBDE congeners).

The chemicals targeted by the ADEC EH Laboratory are: total arsenic, cadmium, chromium, lead, selenium, nickel, methylmercury, and total mercury. EPA methods for the chemical analyses are identified in the below tables and attached SOPs (Appendices B, C, E-G). When specimens are received at the ADEC EH Laboratory, a lab technician will evaluate the fish to ensure that they were properly labeled, packaged and received with sufficient ice to keep them at near-freezing or colder temperature, and that they arrived in good condition. Collection information will be entered into a database and each sample given a unique laboratory identification number. The sample will then be placed in a freezer (temperature range –15ºC to –20ºC or 5ºF to -4ºF). The laboratory technician will record the physical data for each fish (fork length, weight, sex) and collect the otolith for age determination. If a sample is received that is not in adequate condition e.g. received without ice, decomposed, or otherwise physically damaged to compromise the integrity of the sample, the lab technician will make a record to note the disposal of the sample and SVT staff will be contacted to obtain additional replacement fish samples through coordination with partner villages. The cleaning and preparation of all equipment by ADEC staff prior to processing fish tissue will ensure no cross contamination between samples. All knives, cutting boards and grinder parts will be washed with an approved laboratory detergent and rinsed with distilled water. Then all equipment will be rinsed with acetone, hexane, dichloromethane (3 times each) and air dried.

Each fish will be placed on a pre-cleaned cutting board at the lab. Each fish will be cut into strips and homogenized in a grinder. For each village, three whole body fish collected per sampling event will be homogenized into one composite sample (so 36 total individual specimens = 12 composite samples total). Therefore, for each village, two composite samples will result from 6 fish being collected during the 1st sampling event and 1 composite sample from three fish being collected during the 2nd sampling event. **Because three whole body fish (which will vary in size) are being homogenized together into one composite sample, individual specimens will not contribute equally in terms of volume/weight to the composite sample. Homogenization SOPs are included in Appendix E.** The specific steps of homogenization of whole body fish are as follows as stated in Appendix E:

**Commented [KL14]:** Too bad they won't have a few fillet samples to work out correlations between whole body and fillet contaminant levels. Others have done this work though, and the report can use other's work to discuss this relationship.

9.5 Homogenization of Whole Body Fish (if requested).

9.5.1 This section is only followed if whole body analysis is requested by the Project Manager.

9.5.2 After removing the otoliths and preparing the fillet homogenate, grind the remainder of the fish. Whole samples should be homogenized immediately since grinding and homogenization of biological tissue is easier when the tissue is partially frozen. Excess fillet tissue, if available, should be added to the whole fish before homogenization.

9.5.3 Grind the whole sample in a commercial tissue grinder or mince it by hand using the fillet knife. Large samples should be run through the grinder at least three times. A Teflon spatula or tamper tool should be used to ensure that all tissue is continually being run through the grinder blades.

9.5.4 If only small amounts of tissue are available, do not use the grinder. Using the grinder results in loss of mass. The sample should be trimmed to remove possible contaminants and then minced with the fillet knife. The resulting tissue should be roughly comparable to the grinder homogenate.

9.5.5 Homogenates should be divided amongst labeled 4 oz jars depending on needs for analysis. They are frozen and stored at -20°C. Each homogenate portion should be roughly three ounces. The jar should not be completely filled. Analytical decisions are made by the Project Manager.

The homogenate will be divided into 4 portions. Each sample portion (one for trace metals, one for all other contaminants, one for reference, one for potential use as a blind duplicate) will be placed in an approved (certified pre-cleaned) glass sample container (I-Chem jar) labeled with the sample number and frozen at –15ºC to –20ºC or 5ºF to -4ºF. Maximum holding time for frozen samples is one year. The sample portions being sent to the contract lab will be held at the ADEC EH Laboratory until there are a minimum of 15 samples to send. The freezer will be armed with a temperature alarm, and internal temperature in the freezer will be continuously monitored. If the temperature in the freezer moves outside a range of –15ºC to –20ºC or 5ºF to -4ºF an audible alarm will sound in the lab. If after hours, the lab chief or a designated employee will be notified, and will respond appropriately.

Two grinder rinsate samples will be prepared by washing the grinder following standard procedures, and then rinsing de-ionized water over the grinding surfaces and into amber glass bottles. The rinsate samples will be kept refrigerated and included with the tissue samples in the analysis. An ADEC lab blank will also be included with the tissue samples.

Quality Control for the metals and total mercury analyses at the ADEC EH Laboratory includes the analysis of a duplicate for each batch of 10 or fewer samples. For each duplicate a second tissue sample will be removed from one of the jars and analyzed. A random number table will be used to determine which of the samples in the group to use. The results of the duplicate will be compared to the paired sample as a standard quality control measure. The same will be done with the organic analyses, with the exception that a batch at AXYS may contain up to 15 samples. Additionally, every year five blind field duplicates are collected by the ADEC Fish Program. Each blind duplicate is a second set of jars of homogenized tissue from a selected sample. The blind duplicates are given separate Lab Numbers and are treated as original samples for analytical purposes. They are analyzed at the EH Laboratory for metals and at AXYS

for organics, and the data reported to the Fish Program. Fish Program staff will then compare the analytical results of the blind duplicate with the original sample for analytical consistency.

An aliquot of selected homogenized samples will be sent to a contract lab under chain-of-custody for additional chemical analysis. The homogenization/processing for all samples undergoing analyses for organic contaminants, though, will take place at the ADEC EH laboratory. The ADEC EH Laboratory will express ship (overnight delivery) frozen samples to the contract lab (AXYS). The contract lab will be notified via fax or electronically (email) of the impending delivery, along with the tracking numbers. The shipment will include a chain-of-custody document (see Appendix D) and an explanation of what samples were shipped, including identification numbers. If the contract lab does not receive the sample within 24 hours of shipment from the ADEC EH Laboratory, they will contact the Quality Assurance Officer of the ADEC EH Laboratory and report the delay. The ADEC EH Laboratory will also contact the contract lab to confirm receipt of the sample shipment. In the case of delayed receipt the Quality Assurance Officer of the ADEC EH Laboratory will determine if the delay has impacted the integrity of the samples or will affect the quality of the analytical data. If there is any question, the samples will not be analyzed and new samples will be sent. All incidences will be recorded on the chain-of-custody paperwork (see Appendix D).

The contract lab will keep all samples frozen until processed for analysis. All sample material remaining after subsamples are removed for extraction will be refrozen. The contract lab will hold all excess sample material and extracts for one year after the results have been delivered. At that time the contract lab will contact appropriate ADEC EH Laboratory or ADEC personnel to determine whether the samples and extracts are to be discarded or returned to the state. Conditions required for the storage of the samples and extracts at the contract lab are that they be kept frozen at -20˚C or -4˚F and in the dark at all times.

Holding times, for samples to be analyzed, for frozen organic samples is 1 year, extract holding extending another year. For metals the holding time is not well defined but generally 6 months. The contract lab (AXYS) will analyze the samples for selected PCBs and pesticides, following EPA approved methods, while percent lipid will be determined gravimetrically, and PBDEs will be analyzed using a modified EPA method (EPA 1614A). The typical detection limits, method detection limits, low calibration limits, and reporting limits for the organic contaminants are included in Appendix H.

Target Compounds and Detection Limits

| **Parameter** | **Matrix** | **Minimum Detection Limits at 95% Confidence level (ppm)** | **Minimum Reporting Level (ppm wet weight tissue)** |
|---|---|---|---|
| Total Mercury | Whole Body | 0.005 | 0.01 |
| Cadmium | Whole Body | 0.005 | 0.01 |
| Copper | Whole Body | 0.2 | 0.2 |
| Lead | Whole Body | 0.02 | 0.05 |
| Selenium | Whole Body | Undetermined at this time | 0.05 |
| Total arsenic | Whole Body | 0.05 | 0.05 |

| | | | |
|---|---|---|---|
| Organochlorine Pesticides | Whole Body | See Appendix B | See Appendix H |
| PCB Congeners | Whole Body | See Appendix B | See Appendix H |
| PBDE Congeners | Whole Body | See Appendix B | See Appendix H |
| Percent Lipid | Whole Body | | |

1. Percent Lipid

The determination of lipid content in a sample extract is carried out by quantitatively measuring (by weight or by volume) an aliquot of an extract prepared for one of the organic analyses to be performed on the samples, typically either the PCB or dioxin/furan analysis. Each aliquot is placed into a pre-weighed foil weigh boat. The solvent is allowed to evaporate at room temperature prior to drying of the extract at 105°C or 221ºF for 30 minutes. When cool, the weigh boat is re-weighed to determine the weight of lipid. The percent lipid in the sub-sample of extract is determined as the weight of the remaining material divided by the weight of the sample with solvent. The above lipid determination is performed in duplicate and the average percent lipid is reported. The percent recoveries of the labeled surrogate compounds in the remaining extract are corrected for amount of extract consumed in the lipid determination.

2. Trace Metals

Analysis for Arsenic, Cadmium, Chromium, Lead, Selenium and Nickel will be performed by the following method(s). The most recent revision of each method as listed in SW-846 will be used:

| **Analyte** | **Preparatory Method** | **Analytical Method** |
|---|---|---|
| Total Arsenic | EPA Method 3050, 3051 or 3052 | EPA Method 6020 |
| Cadmium | EPA Method 3050, 3051 or 3052 | EPA Method 6020 |
| Copper | EPA Method 3050, 3051 or 3052 | EPA Method 6020 |
| Lead | EPA Method 3050, 3051 or 3052 | EPA Method 6020 |
| Selenium | EPA Method 3050, 3051 or 3052 | EPA Method 6020 |

The preferred method for metals analysis for fish tissues and other environmental matrices is EPA Method 6020, "*Inductively Coupled Plasma - Mass Spectrometry* which ADEC's EH Laboratory utilizes. The use of ICP/MS technology will enable the laboratory to measure the presence of metals in seafood at the lowest possible levels with greater efficiency and savings. The same measurement quality objectives for Trace Metals Analysis as listed in the below tables will be followed. Unless levels of total arsenic are found to be really high, inorganic arsenic is not tested for by ADEC in fish because based upon existing data ADEC has, levels are low.

3. Total Mercury

Total mercury will be determined by EPA Method 7473, Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrometry.

4. Organochlorine Pesticides

29 Organochlorine pesticides will be determined by USEPA Method 1614A. See Appendix H for typical detection limits, method detection limits and low calibration limits.

5. PCB and PBDE Congeners

Analysis for 209 Polychlorinated Biphenyls (PCB) and 46 Polybrominated Diphenyl Ethers (PBDE) congeners will be determined by USEPA Method 1614A. The cleanup techniques described in the method will be employed as necessary to eliminate interferences and to obtain the best possible reporting limits. See Appendix H for typical detection limits, method detection limits and low calibration limits.

## 6.0 QUALITY OBJECTIVES AND CRITERIA

### 6.1 Project Quality Objectives

The primary purpose of this project is to increase basic knowledge and skills in contaminant sampling of fish tissue and collect contaminant data for Sockeye salmon collected within Cook Inlet. By doing this project SVT and SVT staff will build capacity in these areas of collecting contaminant data.

Since collection methods will follow those established by ADEC's Fish Tissue Testing Program and this is a collaborative project with ADEC, project results will be standardized; incorporated into ADEC's databases and on-going research; and shared with university researchers, other state and federal agencies (EPA, NOAA, Department of Interior, ADF&G, DHSS) to further work in evaluating toxicologic impacts on the coastal ecosystem and salmon health issues.

Quality Control methods that will be in place during field collection:

1) Copy of QAPP on board boat
2) Use of labels and labeling
3) Use of Fish Sampling Forms
4) SVT Environmental Coordinator or Assistant serving as QA monitors onsite (for at least the 1st collection event at each village)
5) Fish specimens being immediately "bagged" using nitrile gloves and put into a cooler (with ice) while on board boat
6) Fish specimens being kept away from petroleum products/boat exhaust
7) Once collected, fish specimens are taken immediately back to shore and frozen at -20°C or –4°F before being shipped. Thermometers will be provided and used to ensure proper temperature.
8) Specimens shipped within 24 hours

By following the methodology outlined in this QAPP for collecting fish tissue samples, we can be assured of providing high quality samples to the State for useful and defensible information.

### 6.2 Data Quality Objectives

Since the specimens will be sent to laboratories utilized and/or owned by ADEC, the Quality Assurance (QA) procedures and analytical SOPs and associated laboratory Quality Control (QC) in terms of types & frequencies of QC samples and QC acceptance limits have been determined to be adequate to meet the data quality needs of this project. The analytical methods used by the two laboratories will be EPA Methods or Standard Methods, both well-documented and published methods (see attached Appendices B, C, E-G). The laboratory-established control limits shall be used as acceptance limits for accuracy and precision for this project. The precision and accuracy control limits are listed in the SOPs for the procedures to be used.

AXYS Analytical Services' quality policies meet or exceed ISO 17025 standards. ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories" is an international standard that specifies the management and technical requirements for competence to perform test measurements and calibrations. The ADEC EH Laboratory certifies commercial and municipal laboratories within the State of Alaska to conduct analyses of drinking water and accredits commercial laboratories to conduct analyses including soil remediation.

As such, the laboratories' QC and SOPs (attached as Appendices A, B, C, E, F, G) have been accepted as the project's measurement performance criteria for the analytical component. The laboratories will report detection levels on a sample/analyte-specific basis. Method detection limits (MDLs) will be provided. A copy of the ADEC Environmental Health (ADEC EH) Laboratory's Quality Manual is attached to this QAPP as Appendix A.

**Summary of Measurement quality objectives for Trace Metals Analysis**

| QC Element | Description | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|---|
| Initial Calibration | ICP: 1 std and blank<br><br>GFAA: 3 stds and blank | Daily | <br><br>r > 0.995 | Reanalyze calibration. |
| Instrument precision | ICP: %RSD 3 integrations<br><br>GFAA: RPD of 2 injections | Each calibration and calibration verification | ICP: %RSD < 5%<br><br>GFAA: RPD±10% | Recalibrate and reanalyze |
| Initial Calibration Verification (ICV) | Midlevel ($2^{nd}$ source) verification | After initial calibration. | %Rec= ±10% | Recalibrate and reanalyze. |
| Initial Calibration Blank (ICB) | Interference free matrix to assess analysis contamination. | After initial calibration | All analytes < MDL | Re-calibrate and re-analyze |
| Continuing Calibration verification (CCV) | Midlevel verification | Every 10 samples and at end of analytical sequence. | ICP: %Rec= ±10%<br><br>GFAA: %Rec= ±20% | Recalibrate and reanalyze samples not bracketed by acceptable CCV |
| Continuing Calibration Blank (CCB) | Interference free matrix to assess contamination. | Every 10 samples and at end of analytical sequence. | Analytes < MDL | Reanalyze samples not bracketed by an acceptable CCB |

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|---|---|---|---|---|
| Method Blank (MB) | Interference free matrix taken through entire preparation and analysis process | One per batch, not to exceed 20 samples per batch. | All target analytes must be < one-half of the Reporting Limit (RL). | If blank ≥ one half RL, and all samples ND, no action necessary. If blank < 5% of sample results qualify data. If Blank ≥ one half RL and > 5% of sample results re-extract and re-analyze samples. |
| Laboratory Control Sample (LCS) | Interference free matrix spiked with all target compounds at Midlevel of ICAL | One per batch, not to exceed 20 samples per batch. | %Rec= 80%-120% | If LCS > upper control limit and samples ND, no action needed. Otherwise, re-extract and re-analyze batch. |
| Matrix Spike/ matrix spike duplicate (MS/MSD) | Spike at mid point of ICAL | One MS/MSD per batch, not to exceed 20 samples per batch | Advisory limits<br><br>%Rec= 75%-125%<br>RPD < 25%<br>Evaluate only if spike amount is > 5x sample concentration. | Qualify results. If LCS acceptable and MS/MSD outside limits qualify results as possible matrix effect. |
| Duplicate (Dup) | Laboratory duplicate | One Dup per batch, not to exceed 20 samples per batch | %RPD < 25% | Evaluate results, qualify data. |
| Post digestion spike (PDA) | Sample digestate spiked with target analytes | ICP: 1 per batch.<br><br>GFAA: Every sample | %Rec=75%-125%. | Re-extract and/or re-analyze sample(s). |
| Serial dilution | 1:4 dilution analyzed to assess matrix effects | As needed to assess new matrices. | Agreement between undiluted and diluted results ±10% | Evaluate data, may require dilution and reanalysis of samples. |
| Method of Standard Additions (MSA) | Method of quantitation | As needed for samples with suspected or confirmed matrix effects | r ≥ 0.995 | Evaluate data. |
| Method Detection Limit Study (MDL) | Minimum 7 replicates spiked at 3-5 times the estimated MDL | Prior to analysis of samples, then annually | 40 CFR Part 136, Appendix B | Acceptable MDL must be performed prior to sample analysis. |
| Initial Demonstratio | 4 replicates of LCS | Prior to analysis of samples, then annually | %Rec= 80%-120% | Re-extract and re-analyze IDC. Acceptable IDC |

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| n of Capability (IDC) | | | | must be performed prior to analysis of samples. |
| Performance Evaluation (PE) | Single blind, standard reference material, outside vendor or agency. | Prior to analysis of samples, then annually | Acceptance limits established by PE sample vendor or agency | Determine cause of error. Re-analyze new PE sample. Acceptable PE must be performed prior to analysis of samples. |

**Summary of Measurement quality objectives for Mercury by EPA Method 7473**

| QC Element | Description | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|---|
| Initial Calibration (ICAL) | Low range: 4 standards and blank<br><br>High range: 6 standards and blank | Prior to sample analysis, then as required | r > 0.995 | Reanalyze calibration. |
| Initial Calibration Verification (ICV) | Midlevel (2nd source) verification. Includes at least a high and low concentration standard for each working range. | After initial calibration | %Rec= ±10% | Recalibrate |
| Daily Calibration | Midlevel verification includes at least a high and low concentration standard for each working range. | Daily, after every 10 samples and at end of analytical sequence. | %Rec= ±10% | Recalibrate and reanalyze samples not bracketed by acceptable Daily Calibration |
| Method Blank (MB) | Interference free matrix taken through entire preparation and analysis process | One per batch, not to exceed 20 samples per batch. | All target analytes must be < one-half of the Reporting Limit (RL). | If blank ≤ one half RL, and all samples ND, no action necessary. If blank is > 5% of sample results qualify data. If blank ≥ one half RL and > 5% of sample results re-extract and re-analyze samples. |
| Laboratory Control Sample (LCS) | Interference free matrix spiked with all target compounds at Midlevel of ICAL | One per batch, not to exceed 20 samples per batch. | %Rec= 80%-120% | If LCS > upper control limit and samples ND, no action needed. Otherwise, re-extract and re-analyze batch. |
| Matrix Spike/ matrix spike duplicate (MS/MSD) | Spike at mid point of ICAL | One MS/MSD per batch, not to exceed 20 samples per batch | Advisory limits<br><br>%Rec= 80%-120%<br>RPD < 20%<br>Evaluate only if spike amount | Qualify results. If LCS acceptable and MS/MSD outside limits qualify results as possible matrix effect. |

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| | | | is > 5x sample concentration. | |
| Duplicate (Dup) | Laboratory duplicate | One Dup per 10 samples or fraction of 10 when the batch is greater than 10 | %RPD < 25% | Evaluate results, qualify data. |
| Method of Standard Additions (MSA) | Method of quantitation | As needed for samples with suspected or confirmed matrix effects | r ≥ 0.995 | Evaluate data. |
| Method Detection Limit Study (MDL) | Minimum 7 replicates spiked at 3-5 times the estimated MDL | Prior to analysis of samples, then annually | 40 CFR Part 136, Appendix B | Acceptable MDL must be performed prior to sample analysis. |
| Initial Demonstration of Capability (IDC) | 4 replicates of LCS | Prior to analysis of samples, then annually | %Rec= 80%-120% | Re-extract and re-analyze IDC. Acceptable IDC must be performed prior to analysis of samples. |
| Performance Evaluation (PE) | Single blind, standard reference material, outside vendor or agency. | Prior to analysis of samples, then annually | Acceptance limits established by PE sample vendor or agency | Determine cause of error. Re-analyze new PE sample. Acceptable PE must be performed prior to analysis of samples. |

**Summary of Measurement quality objectives for USEPA Method 8081**

| QC Element | Description | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|---|
| Initial Calibration (ICAL) | Minimum 5 points, low level at or below reporting limit | Prior to sample analysis | r $\geq$ 0.995 or $r^2 \geq$ 0.990 or %RSD $\leq$ 20% | Recalibrate |
| Initial Calibration Verification | Midlevel ($2^{nd}$ source) verification | After each new ICAL. | %Rec= 85%-115% | Check for problems with ICAL and or second source. Reanalyze ICAL. |
| Continuing Calibration Verification | Midlevel verification | At beginning and end of each 12 hour analytical shift; after every 10 samples and at the end of the analytical sequence whichever is most frequent. | %Drift = 15%, or %D < 15% | Check for problems; reanalyze CCV. If unacceptable after $2^{nd}$ injection recalibrate. Samples not bracketed with an acceptable Calibration verification must be reanalyzed. |
| Method Blank (MB) | Interference free matrix taken through entire preparation and analysis process | One per batch, not to exceed 20 samples per batch. | All target analytes must be < one-half of the Reporting Limit (RL) | If blank $\geq$ one half RL, and all samples ND, no action necessary. If blank < 5% of sample results qualify data. If |

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|---|---|---|---|---|
| | | | | Blank ≥ one half RL and > 5% of sample results re-extract and re-analyze samples. |
| Laboratory Control Sample (LCS) | Interference free matrix spiked with all target compounds at Midlevel of ICAL | One per batch, not to exceed 20 samples per batch. | %Rec= 50%-130% or in house generated limits. | If LCS > upper control limit and samples ND, no action needed. Otherwise, re-extract and re-analyze batch. |
| Matrix Spike/ matrix spike duplicate (MS/MSD) | Field sample spiked with all target compounds at Midlevel of ICAL | One MS/MSD per batch, not to exceed 20 samples per batch | Advisory limits: %Rec= 50%-130% or in house generated limits.<br><br>RPD < 50%<br>Evaluate only if spike amount is > 5x sample concentration. | Qualify results. If LCS acceptable and MS/MSD outside limits qualify results as possible matrix effect. |
| Duplicate (Dup) | Laboratory duplicate | One Dup per batch, not to exceed 20 samples per batch | %RPD < 50% | Evaluate results, qualify data. |
| Surrogate spikes (Surr) | | Every MB, LCS, MS, MSD, Dup, and sample | %Rec=50%-130% or in house generated limits. | If %Rec is outside limits for MB or LCS re-extract and re-analyze entire batch. If %Rec > upper control limit in a sample and sample is ND for all compounds, no action. Otherwise, re-extract and re-analyze samples with surrogate outside limits. |
| Method Detection Limit Study (MDL) | Minimum 7 replicates spiked at 3-5 times the estimated MDL | Prior to analysis of samples, then annually | 40 CFR Part 136, Appendix B | Acceptable MDL must be performed prior to sample analysis. |
| Initial Demonstration of Capability (IDC) | 4 replicates of LCS | Prior to analysis of samples, then annually | %Rec= 50%-130% or in house generated limits. | Re-extract and re-analyze IDC. Acceptable IDC must be performed prior to analysis of samples. |
| Performance Evaluation (PE) | Single blind, standard reference material, outside vendor or agency. | Prior to analysis of samples, then annually | Acceptance limits established by PE sample vendor or agency | Determine cause of error. Re-analyze new PE sample. Acceptable PE must be performed prior to analysis of samples. |

| Summary of Measurement quality objectives for USEPA Method 1668B* | | | | |
|---|---|---|---|---|
| **QC Element** | **Description** | **Frequency** | **Acceptance Criteria** | **Corrective Action** |
| Initial Calibration (ICAL) | Minimum 5 points | Prior to sample analysis | Per method 1668A | Recalibrate |
| Calibration Verification | Midlevel verification | At beginning and end of each 12 hour analytical shift | Per method 1668A | Per method 1668A |
| Method Blank (MB) | Interference free reference matrix taken through entire preparation and analysis process | One per batch, not to exceed 20 samples per batch. | Per method 1668A | Per method 1668A |
| Ongoing Precision and Recovery Sample (OPR) | Interference free reference matrix spiked with all target compounds at Midlevel of ICAL | One per batch, not to exceed 20 samples per batch. | Per method 1668A | Per method 1668A |
| Duplicate (Dup) | Laboratory duplicate | One Dup per batch, not to exceed 20 samples per batch | %RPD < 50% | Evaluate results, qualify data. |
| Surrogate spikes (Surr) | | Every sample and QC sample. | Per method 1668A | Per method 1668A |
| Method Detection Limit Study (MDL) | Minimum 7 replicates spiked at 3-5 times the estimated MDL | Prior to analysis of samples, then annually | 40 CFR Part 136, Appendix B | Acceptable MDL must be performed prior to sample analysis. |
| Initial Precision and Recovery (IPR) | 4 replicates | Prior to analysis of samples, then annually | Per method 1668A | Re-extract and re-analyze IPR. Acceptable IPR must be performed prior to analysis of samples. |
| Performance Evaluation (PE) | Single blind, standard reference material, outside vendor or agency. | Prior to analysis of samples, then annually | Acceptance limits established by PE sample vendor or agency | Determine cause of error. Re-analyze new PE sample. Acceptable PE must be performed prior to analysis of samples. |

*Additional Quality Control criteria are included in the analytical method.

## 7.0 DELIVERABLES, DATA STORAGE AND ANALYSIS

Once the fish and completed sample data forms are received at the ADEC EH Laboratory, a lab technician will enter the data into a SQL Server database. A unique sample tracking number will be assigned to each sample at that time. The sample number is assigned as: year, village, sampling event #/period, and fish #. As examples, for Seldovia, the sample number assigned to fish would be 14SE0101, 14SE0102, 14SE0103, etc. for the 1st sampling event and the second sampling event would be 14SE0201, 14SE0202, 14SE0203, etc. Sample collection data sheets, chain-of-custody forms, sample tracking forms, and bench sheets containing calculations will also

be filed at the ADEC EH Laboratory. As analyses are performed, raw or calculated results will be added to the database (this will depend on machine output). When subsamples are sent to the contract lab (AXYS), a chain-of-custody form (see Appendix D) will be sent with it, providing a tracking number for the sample. The contract lab will send an electronic copy of the results (a read only file) of all of its analyses back to the ADEC EH Laboratory, with all data referenced by the sample tracking number. A second copy of the results will be sent to a third party contractor for data validation, results of which will be provided as both electronic (read only file) and hard copy files. The electronic data files from the contract lab and the data validation contractor will be downloaded directly into the ADEC EH Laboratory database. The validated data will be used to generate the program reports. Once the data has been validated, a hard copy and electronic copy (as a read only file) will be sent to both EPA Region 10 (as part of the deliverables) and to Seldovia Village Tribe staff, who will then share the data with partner tribes (Port Graham, Tyonek, and Nanwalek). Throughout the study the ADEC EH Laboratory Quality Assurance Officer and ADEC Fish Tissue Program Coordinator will check fish sample processing and the dataset for errors by comparing sample processing and data entry sheets with the results in the electronic database.

Hard copy documents to be retained at the ADEC EH Laboratory include original chain-of-custody documents (this includes log information), sample metadata (age, sex, length, weight, any abnormalities), in-lab sample tracking forms, sample analytical results of the required analyses, and graphic results of the analyses; the electronic data include sample log-in, data generated during analysis, quality control data, and the final analytical data.

Data deliverables required from all laboratories (to be used for data validation) includes sample analytical results, blind and laboratory duplicates, MS/MSDs blanks, and calibration checks, in addition to the required storage of the electronic raw data generated in both the ADEC EH Laboratory and the contract lab. None of the data will be purged by the labs without prior authorization from the ADEC EH Director.

Data, once received by SVT staff, will be kept on SVT office computers and combined data entered into an excel database (if necessary). SVT office doors are locked every night and computers are password protected and backed up offsite to ensure data longevity and security.

*Data Analysis*

The basic data analysis will be performed with a commercial statistical software program (SPSS Inc.) by ADEC staff. Mean, standard deviation, and median values will be calculated for contaminant concentrations in each species sampled. If sufficient data is available, mean, standard deviation, and median values will also be calculated for groupings within a species: sex, age length, and weight. Analysis of variance and analysis of co-variance may be calculated for contaminant loads among general collection locations for species where there are sufficient data points and the basic assumptions for data quality are met. If the Data quality assumptions are not met, non-parametric alternatives will be used in the analysis.

## 8.0 ASSESSMENT AND RESPONSE

Assessment policies of the ADEC EH Laboratory are included in Appendix A. The ADEC EH Laboratory maintains a program for internal assessments of its quality management system and operations, and participates in external assessments such as proficiency testing and regulatory

and accreditation inspections. These programs help verify that ADEC EH Laboratory operations continue to comply with quality system requirements and requirements of applicable standards and regulations.

The ADEC EH Laboratory assesses all areas of the management systems at least once every three years. The Quality Systems Manager (QSM) is responsible for planning assessments. Unscheduled assessments may take place if the QSM identifies a need. The ADEC EH Laboratory uses trained, in-house personnel independent of the activity to be assessed (whenever resources permit) or qualified third parties to conduct internal assessments.

The ADEC EH Laboratory maintains a documented procedure that addresses internal assessment criteria and scope, frequency and methodologies, responsibilities, and requirements for personnel conducting the assessments.

When internal assessment findings cast doubt on the effectiveness of the operations or on the correctness or validity of test results, the lab takes corrective action in a timely manner.

The QSM is responsible for tracking corrective actions necessary to satisfy deficiencies found during the assessment. The corrective actions are documented in the same manner as nonconformances.

The QSM monitors follow-up actions to verify and record the implementation and effectiveness of the corrective actions taken.

The ADEC EH Laboratory participates in the periodic external inspections and assessments that are required to maintain its accreditation or certification status.

The laboratory participates in the triennial onsite laboratory evaluation programs conducted by federal officers, to include EPA Safe Drinking Water Act (SDWA) laboratory certification officers, FDA laboratory evaluation officers, or FDA certified state laboratory evaluation officers.

The laboratory participates in ongoing inspections, assessments, and performance evaluation sample analysis sponsored by various agencies, including but not limited to:

- USDA-APHIS (Approval for EIA, Brucellosis analysis)
- FDA (Dairy, Shellfish)
- EPA (Drinking water)
- OSHA

When external assessment findings cast doubt on the effectiveness of the operations or on the correctness or validity of ADEC EH Laboratory test results, the lab takes corrective action within the deadlines set by the certifying agency. The laboratory participates in annual proficiency test programs appropriate for its testing capabilities. Examples include, but are not limited to, FDA-sponsored splits testing, and Water Supply Proficiency Testing required by the EPA Safe Drinking Water Program. The QSM maintains records of internal assessments, including follow-up actions to correct identified deficiencies. The QSM maintains records of external inspections and assessments, and the follow-up actions to correct identified deficiencies. Results of internal assessments and external assessments are provided for management review. Management

reviews internal and external assessment results at each management review meeting. The ADEC EH Laboratory management ensures that appropriate communication processes are established within the laboratory and that communication takes place regarding the effectiveness of the management system.

**9.0 DATA VALIDATION AND USABILITY**

Data review policies of the ADEC EH Laboratory are included in Appendix A. Where appropriate, the laboratory uses Statistical Quality Control (SQC) to monitor the consistency and accuracy of testing procedures. The Quality Assurance Manager monitors trends, evaluates SQC data, and establishes internal Quality Control limits, where appropriate. The quality control check data are analyzed and, where they are found to be outside pre-defined criteria, planned actions are taken to resolve the problem or anomaly. Intentional deviation from test methods occur only if the deviation has been documented, technically justified, and authorized. The client is notified, and client acceptance is sought when appropriate. Unintentional deviations are documented as nonconformances.

The laboratory uses a multi-level review process to ensure the quality of final results submitted to the client. Analytical results are reviewed for compliance with SOP requirements, data entry accuracy, report format, and client requirements. Analytical results are not released without review by at least two separate persons (analyst and 2nd level review by a supervisor or peer). This review process is described in detail in the Quality Process (QP)-18 Results Review. The ADEC EH Laboratory notifies the client in writing of any typographical error(s) or of any defective test equipment that casts doubt on the validity of results stated in the report. The ADEC EH Laboratory amends or corrects test reports after issue in a manner as to refer to the original report it replaces. Staff are not authorized to give opinions concerning the meaning of client results.

The ADEC EH Laboratory maintains a documented procedure that addresses the activities implemented whenever any testing activity, or the results of such activity, does not conform to ADEC EH Laboratory procedures or testing requirements.

Records are maintained of all investigations and corrective actions taken by the laboratory.

The procedure for control of non-conforming tests:

- defines non-conforming tests;
- designates responsibilities and authorities for managing nonconforming work;
- specifies possible actions taken when nonconforming work is identified;
- requires an investigation to determine why work is nonconforming;
- provides for the evaluation o f the significance of the nonconforming work;
- indicates that corrective actions are required immediately;
- requires a decision regarding the acceptability of the nonconforming work;

- addresses client or other appropriate notification activities;
- indicates how nonconforming work is to be recalled if appropriate; and
- defines the responsibilities for authorizing the resumption of work.

The ADEC EH Laboratory notifies the client in writing of any typographical error or any defective measuring or test equipment that casts doubt on the validity of the results stated in the report.

**10.0 REPORTING AND OUTREACH**

Laboratory results will be sent directly to ADEC staff involved in the Fish Tissue Testing Program, who will then compile and analyze those results. Electronic files containing results will be sent to SVT from ADEC. Upon receiving results, SVT will share the results with all partner Tribes, the Tribal Council, and EPA. A project summary report will be developed by SVT Environmental Personnel and sent out to all the above parties as well.

Additionally, SVT's Environmental Coordinator will submit quarterly reports to our IGAP Project Officer to keep EPA informed of project progress.

**11.0 REFERENCES**

[ADEC] Alaska Department of Environmental Conservation Fish Monitoring program. Available online at http://www.dec.state.ak.us/eh/vet/fish.htm

[ATSDR] Agency for Toxic Substances and Disease Registry. 2009. Evaluation of seafood and plant data collected from Cook Inlet near the native villages of Port Graham, Nanwalek, Seldovia, and Tyonek, Alaska. Atlanta, Georgia

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Assessment of contaminant body burdens and histopathology of fish and shellfish species frequently used for subsistence food by Alaskan Native communities. Available online at http://doc.nprb.org/web/10_prjs/1019_Final%20report.pdf

[SVT] Seldovia Village Tribe. 2013. Assessment of Cook Inlet Tribes Subsistence Consumption. Seldovia, Alaska.